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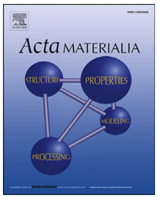

Full length article

Pre- and post-buckling behavior of bi-crystalline micropillars: Origin and consequences

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ABSTRACT

Compression of micropillars is routinely used to measure the material response under uniaxial load. In bi-crystalline pillars an S-shaped grain-boundary together with an S-shaped pillar is often observed after deformation raising the question of its origin and consequences for stress-strain materials data. In addition to dislocation and grain-boundary based mechanisms, this observation can be caused by buckling and subsequent post-buckling deformation. Deviations from the classical pre- and post-buckling deformation behavior are assigned to imperfections, which are categorized in extrinsic and intrinsic imperfections in this work. In the present paper, the S-shaped actual deformation state is particularly promoted by an intrinsic imperfection, caused by a material heterogeneity (due to the bi-crystal arrangement). This kind of deformation behavior is investigated by micro-compression experiments on 7 × 7 × 21 μm^3 sized bi-crystal copper pillars with nearly elastic (axial Young's modulus) homogeneity and identical Schmid factors for both grain orientations. Complementary finite element simulations are performed, in which also the role of friction and of an extrinsic imperfection in the form of initial misalignment of the loading on the S-shape are considered. There, a material model describing the flow stress distribution caused by a dislocation pile-up at the grain-boundary is applied. Finally, suggestions to prevent buckling and, thus, transversal post-buckling displacements during micropillar compression tests are given with the goal to extract engineering stress-strain curves.

1. Introduction

Micropillar compression, as pioneered by Uchic and co-workers [1], is routinely applied for performing uniaxial loading experiments at the micron and sub-micron scale. The experiments were initially conducted to ensure a uniaxial stress state and to provide material parameters at mesoscopic length scales. Unexpectedly, a size effect of the flow stress was observed even in the absence of strain gradients [2–4]. Today it is evident that the number of dislocation sources and their sizes play a dominant role and result in a transition from a deterministic to a stochastic behavior in smaller dimensions [5,6].

The role of this sample size effect on the deformation behavior of bulk materials is not yet fully clear. Thus, during the last years several studies were aiming to involve one degree more of complexity by including a single grain-boundary [7–13]. The studies were performed on aluminum [7,8,13], nickel [9,10,14] as well as copper [11,12,15,16] bi-crystals. All of the investigated metals have different elastic anisotropy, stacking fault energies, crystal orientations and grain-boundary characters. It is, therefore, not surprising that general statements among all of the studies are still scarce. The only but seldomly explicitly stated finding is the fact that all samples containing non-$\Sigma 3$ grain-boundaries show non-planar grain-boundaries after the deformation. Two cases can be distinguished: (i) a motion of the grain-boundary within the micropillar volume by, e.g. shear-coupled boundary motion, laying down an array of dislocations at the grain-boundary, and diffusion controlled processes, and (ii) a S-shape of the entire micropillar. This work aims for understanding the S-shape of micropillars caused by the post-buckling displacement behavior during

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compression of micropillars. Consequently, this study helps finding well-performed bi-crystalline micropillar experiments which allow for interpreting the engineering stress-strain curves as material response, and ruling out experiments which predominantly reflect the post-buckling displacement behavior (DB), where an engineering stress-strain curve does not make any sense. In the context of this paper, the notation post-buckling is used for the behavior of the compressed micropillar in the deformation process following the instant of buckling, i.e. beyond the load at which the transversal displacement suddenly starts to grow.

Within this study, possible imperfections present in a bi-crystalline micropillar are categorized and critically discussed aiming for extracting "reliable" material properties from bi-crystalline micropillar compression experiments.

2. Classification of imperfections

Two different kinds of imperfections are defined: (i) extrinsic imperfections, caused by experimental uncertainties, and (ii) intrinsic imperfections, originating from material inhomogeneity. Thus, extrinsic imperfections can – in a perfect experiment – entirely be suppressed, whereas intrinsic imperfections are inherent to the tested material and cannot be circumvented.

(i) Extrinsic imperfections include, but are not limited to:
- load imperfections, where the load is not applied in the center of contact or provokes not only purely axial deformation due to an inclination of the load orientation;
- a non-perfect prismatic shape of the pillar (e.g., slightly curved pillar axis);
- a miscut of the micropillar top surface with respect to the flat-punch indenter surface. this is further denoted as "misalignment", see also [17–19];
- a grain-boundary being not vertical in the bi-crystalline micropillar;
- a non-axisymmetric material base, e.g. caused by focused ion beam (FIB) drift during milling.

Due to their nature, most extrinsic imperfections do not depend on whether a single or bi-crystalline sample is tested. Their important role has well been described previously when single crystalline micropillar compression was extensively used to study size effects [17,18].

(ii) Intrinsic imperfections can, for instance, be caused by:
- spatially non-constant Young's moduli, e.g. in a bi-crystalline sample;
- differences in flow stress across the micropillar cross-section;
- formation of slip steps, in particular for single slip oriented crystals, leading to a non-symmetric load transfer along the micropillar height;
- deformation along unstable crystallographic directions, where the crystallographic compression axis changes due to lateral constraints, such as shown by Raabe et al. [17] for single crystals.

Due to a mismatch in Young's moduli the stress state in both grains of a bi-crystalline micropillar can significantly deviate from a homogenous stress state already in the elastic deformation range. Even a secondary stress state (i.e. local stresses in addition to primary stresses originating from the external load) may develop due to local compatibility constraints. This was described by Tiba and co-workers [14] analytically for nickel micropillars, showing the activation of otherwise inactive slip systems. Also, the load is not applied in a centrosymmetric manner (excentric loading with respect to the center of the pillar top-surface) in this case, which is caused by an intrinsic imperfection of the system. Unfortunately, elastic anisotropy is omnipresent in the aforementioned bi-crystalline micropillar studies [7–10,12,13].

Even if elastically homogeneous straining occurs, inhomogeneous plastic strain distributions may exist [20], resulting in an inhomogeneous stress field. For instance, if the Schmid-factors of the two adjacent grains are not identical, the stress during plastic flow is not equally distributed, which again leads to excentric loading upon plastic flow. Even in the case, where the Schmid factors of the two grains are identical, their hardening behavior is likely to be different, which is a further source of a plastic imperfection.

The majority of bi-crystals either shows elastic, or plastic, or even both inhomogeneities, with one exception: the coherent $\Sigma 3(111)$ twin boundary of Imrich and co-workers [12]. Surprisingly, this study is – to the best of our knowledge – the only one where an S-shaped post-deformation geometry can be excluded.

Counterintuitively, friction does not represent an imperfection as it does not necessarily lead to an eccentric load application. Thus, we do not define friction as an imperfection, even though it plays a dominant role during deformation [21], in particular deformation in the post-buckling DB. Hence, a systematic investigation addressing the importance of imperfections cannot be performed without discussing the stabilizing effect of lateral forces caused by friction.

The S-shaped post-deformation geometry could be a simple criterion for the experimentalist whether or not the measured micropillar response is dominated by the post-buckling DB triggered by imperfections or, in other words, if the micropillar response is a good measure for the integral material response. Thus, the aim of this paper is to help finding well-performed bi-crystalline micropillar experiments allowing that the engineering stress-strain curve can be taken as the material response, and ruling out experiments which predominantly reflect the post-buckling DB, where an engineering stress-strain curve does not make any sense. For this purpose we have chosen a sample configuration (arrangement of the crystallographic orientations of the two grains) which is nearly homogeneous in the axial Young's moduli and in Schmid-factors, but likely to be different in the hardening behavior of the two adjacent grains. Finite element modelling (FEM) is used to investigate the important role of friction, intrinsic and extrinsic imperfections.

3. Experimental details

3.1. Sample preparation and testing

$7 \times 7 \times 21\ \mu m^3$ sized copper bi-crystalline micropillars were produced following the procedure of Moser et al. [22]. Thereby, a 3 mm sized platelet is ground to a final thickness of 300 μm and subsequently etched to a wedge with an opening angle of roughly 30°. This procedure enables well defined micropillar geometries with constant cross-sections across the entire micropillar height (see Fig. 1a). However, the thinned region of the lamella increases the lateral compliance of the wedge, which has to be taken into account in FEM modelling. The final micropillar as presented in Fig. 1b is produced by FIB milling with a final polishing current of 500 pA. The crystallographic compression axes of the milled micropillar are shown in Fig. 1a.

As loading rig, a custom-made loading device with a nominal force resolution higher than 10 μN and a nominal displacement resolution of 1 nm was used [23]. The machine works in true displacement controlled mode with a spring constant at the load